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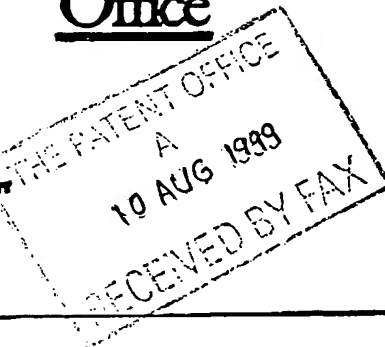
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Your reference

CRO 50828/CB/P

Patent application number

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9918782.5**Full name, address and postcode of the or of
each applicant (underline all surnames)**Crosfield Limited
4 Liverpool Road
Warrington
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United Kingdom**Patents ADP number (if you know it)**

743227201

**If the applicant is a corporate body, give the
country/state of its incorporation**

UNITED KINGDOM

Title of the invention

CLEANING COMPOSITIONS

Name of your agent (if you have one)

COLLINGWOOD, Anthony Robert

John Derek Jackson

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to which all correspondence should be sent
(including the postcode)**ICI GROUP INTELLECTUAL PROPERTY Crosfield Limited
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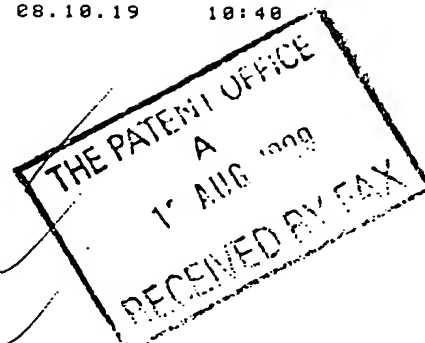
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Abstract

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CLEANING COMPOSITIONS

This invention relates to cleaning compositions and, in particular, particulate cleaning compositions to be formed into moulded bodies for use in for example fabric washing, dishware washing, stain removal and water softening.

Detergent compositions in the form of tablets are widely described and are currently enjoying increasing popularity with consumers. They are described, for example, in GB 911204 (Unilever), US 3953350 (Kao), DE 19637606 (Henkel), EP 711827 (Unilever) and WO 98/40463 (Henkel). Tablets for machine dishwashing applications are described in, for example WO 96/28530 (P&G). Tableted detergents and cleaning compositions have several advantages over powdered or liquid compositions: they are easier to dispense and handle, do not require measuring to obtain the correct dose and, being compact, are more economic to store and transport.

Tablets of cleaning compositions are generally made by compression or compaction of a quantity of the composition in the form of particles. Production of tablets which are sufficiently hard and strong to withstand storage and handling requires a relatively high pressure to be used in this compaction process. It is then necessary that, despite this compaction, the tablets are able to disperse and dissolve rapidly when added to wash water.

One approach to achieving good dispersion of the tablet is to include in the tablet a particulate insoluble but water-swellaable agent. These particles then swell with ingress of water, leading to stresses in the tablet and thence to break-up of the tablet. Thus WO 98/55583 (Unilever) describes the use of 'water-insoluble, water-swellaable polymeric material' which 'promotes disintegration of the tablets in water'. Typical swelling agents which have been disclosed as possible tablet disintegrating agents are starches, cellulose and cellulose derivatives, alginates, dextrans, cross-linked polyvinyl pyrrolidones, gelatines, formaldehyde casein as well as a wide variety of clay minerals and certain ion-exchange resins.

These water swelling agents have no function in fabric washing except to aid tablet disintegration. Furthermore, because they are insoluble and of relatively large particle size, they tend to deposit on clothes during the wash (see for example WO 98/55575 (Henkel) and WO 98/55582). As a result, several attempts have been made to minimise the deposition of these disintegrants, for example by combining such a water-swellaable, insoluble disintegrant with a second, highly soluble disintegrating aid - see WO 98/55582 (Unilever). Other attempts have included use of a preferred particle size of the disintegrant. Thus, for example, WO 98/55583 (Unilever) claims use of such material at a particle dimension of at least 400 microns to give more efficient disintegration. On the other hand, WO 98/55575 (Henkel) teaches the use of cellulose disintegrating aids with a particle size of less than 100 microns, in order to minimise deposition. This material is co-granulated with 'microcrystalline cellulose and/or one or several ingredients of detergents and cleaning agents'.

50828/GB

Surprisingly, we have now found that if a water-swellaible disintegrating aid is co-granulated with an insoluble or sparingly soluble, hydrophilic solid before being incorporated into a tablet, then the disintegrant is much more efficient in disintegrating the tablet in use. Thus less of the disintegrating aid needs to be added for effective disintegration, lowering the probability of deposition of the disintegrating aid on the substrate being cleaned.

The present invention seeks to provide a particulate cleaning composition in the form of a moulded body and which is relatively inexpensive to manufacture and sufficiently robust to withstand handling during production and packaging processes but readily breaks up and dissolves when contacted with an aqueous medium during the cleaning process for which it is intended without resulting in the undue deposition on the substrate being cleaned.

According to the present invention there is provided a cleaning composition, the composition including disintegrant in the form of granules comprising a water insoluble inorganic material and a water-swellaible agent which, in its anhydrous state, comprises no more than 20% of the combined weight of said inorganic material and said agent of the granular disintegrant, the granules being combined with the active ingredients of the cleaning composition in a compacted moulded body.

By "water insoluble", we mean a compound with a water solubility of less than 5 grams, preferably less than 1 gram, per 100 grams water (at a temperature of 25°C?).

Preferably the water-swellaible agent comprises, in its anhydrous state, no more than 15%, more preferably no more than 10%, of the combined weight of said inorganic material and said agent of the granular disintegrant. In a typical composition of the invention, the water-swellaible agent comprises, in its anhydrous state, no more than 8%, e.g. 7.5% or less, of the combined weight of said inorganic material and said agent.

A feature of the invention is the relatively small amount of water-swellaible agent that may be employed while securing satisfactory properties for the compacted cleaning composition.

In one embodiment of the invention the inorganic material used in the formation of the disintegrant comprises a silica.

In a second embodiment of the invention the inorganic material used in the formation of the disintegrant conveniently comprises a material which acts as a functional ingredient of the cleaning composition. In this instance, the inorganic material may be an aluminosilicate such as zeolite P, A or X or mixtures thereof, with zeolite P being preferred.

Alkali metal aluminosilicates, especially zeolites, are commonly used in detergent compositions as a detergency builder. Where a cleaning composition of the invention is one containing an alkali metal aluminosilicate as detergency builder, preferably at least part of the aluminosilicate constituent of the cleaning composition is employed as the inorganic material used in the formation of the disintegrant granules. For instance, the aluminosilicate constituent

CP 50828/GB

typically comprises about 10 to about 60% by weight of the cleaning composition and preferably at least 0.5% of the zeolite constituent is embodied in the disintegrant granules.

The water-swellaable agent conveniently has a primary particle size of no more than 200 microns, preferably no more than 100 microns, and a water swelling capacity of at least 5ml/gram, preferably 10 ml/gram and more preferably 20ml/gram.

Typically the water-swellaable agent comprises a wholly or partially cross-linked polymer e.g. cross-linked cellulose or (sodium) carboxy-methyl cellulose, cross linked starch, or cross linked poly vinyl pyrrolidone. Currently preferred are are Aquasorb A500 (ex Hercules) and Ac-Di-Sol (ex FMC Corp).

The moulded body formed using the cleaning composition of the present invention may consist wholly of the cleaning composition or alternatively the moulded body may comprise a number of discrete portions, at least one of which comprises a cleaning composition in accordance with the invention. In this event, the remaining portion or portions of the moulded body may be constituted by at least one other ingredient, usually one suitable for use in detergent and cleaning applications such as fabric washing, dishware washing, stain removal and water softening.

Cleaning compositions of the invention may also contain, as essential ingredients, one or more detergency builders (wholly or partly incorporated in the disintegrant granules), and/or one or more detergent-active compounds which may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic detergent-active compounds, and mixtures thereof and/or other conventional additives.

Alkali metal aluminosilicates are favoured as environmentally acceptable water-insoluble builders, e.g. zeolites A, X and P or mixtures thereof. Other inorganic detergency builders include layered sodium silicate as described in US-A-4664839 and marketed by Hoechst as SKS-6, and alkali metal (generally sodium) carbonate. Water-soluble phosphorous containing detergency compounds such as alkali metal orthophosphates, metaphosphates, pyrophosphates and polyphosphates may be used. Also possible are organic detergency builders such as polycarboxylate polymers, e.g. polyacrylates, acrylic/maleic polymers, and acrylic phosphonates, monomeric polycarboxylates, e.g., gluconates, oxydisuccinates, glycerol mono- di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates and hydroxyethyliminodiacetates.

Many suitable detergent-active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

The preferred detergent-active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds. Anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene

CF 50828/GB

sulphonates having an alkyl chain length of C sub 8 - C sub 15 ; primary and secondary alkyl sulphates, particularly sodium C sub 12 - C sub 15 primary alcohol sulphates; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C sub 12 - C sub 15 primary and secondary alcohols ethoxylated with an average of from 3 to 20 moles of ethylene oxide per mole of alcohol.

The choice of surfactant, and the amount present, will depend on the intended use of the detergent composition. For example, for machine dishwashing a relatively low level of a low-foaming nonionic surfactant is generally preferred. In fabric washing compositions, different surfactant systems may be chosen, as is well known by the skilled detergent formulator, for handwashing products and for machine washing products.

The total amount of surfactant present will of course depend on the intended end use and may be as low as 0.5% by weight, for example in a machine dishwashing composition, or as high as 60% by weight, for example in a composition for washing fabrics by hand. For fabric washing compositions in general, an amount of from 5 to 40% by weight is generally appropriate.

A suitable type of cleaning composition suitable for use in most automatic fabric washing machines contains both anionic and nonionic surfactants. Cleaning compositions according to the invention may also suitably contain a bleach system. Machine dishwashing compositions may suitably contain a chlorine bleach, while fabric washing compositions may contain peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, which may be employed in conjunction with activators to improve bleaching action at low wash temperatures. Again, the skilled detergent worker will have no difficulty in applying the normal principles to choose a suitable bleach system.

Other materials that may be present in detergent compositions of the invention include sodium silicate, fluorescors, antiredeposition agents, inorganic salts such as sodium sulphate, enzymes, lather control agents or lather boosters as appropriate, pigments, and perfumes. This list is not intended to be exhaustive.

Procedures and Tests

Granule Production

The granules of inorganic material and swelling agent may be prepared by any of the methods that will be known to those skilled in the art, e.g. by blending the dry ingredients in a mixer (such as a Pek mixer available from George Tweedy & Co of Preston - 281b S.A. Machine) and compacting on a roller compactor (Alexanderwerk WP60 - manufactured by Alexanderwerk AG, D 5630 Remscheid 1, Germany). A typical preparative method is now described in detail with reference to silica as the inorganic material. Silica and water swelling organic particulate are blended together, in the appropriate proportions, in a Pek mixer for 30

CF 50828/GB

minutes. A minimum of 2 kg of blended material so prepared is compacted by feeding into an Alexanderwerk roller compactor, fitted with a sintered block vacuum deaeration system. The roller pressure setting is selected according to the strength of granule desired, higher pressures leading to stronger granules. A typical roller pressure would be 100 bar.

The compacted material from the compactor is fed into a granulator, which forms part of the machine, and forced through a mesh and the resulting granules are then screened to the desired particle size range, e.g. an average particle size of 250 to 750 microns, using standard laboratory sieves.

Tablet Production

Tablets used in the Examples that follow were produced using a 45 mm diameter die set (stainless steel) in conjunction with a Universal Testing Machine Type No. Z030 from Zwick GmbH, Ulm, Germany. A known quantity, 40-45grams, of the cleaning composition is placed in the die, the die plunger is inserted and the assembly is placed between the platens of the Zwick machine which is operated to apply a predetermined pressure to produce a tablet having a defined density and, in particular a dimensionally stable and fracture resistant tablet. Tablets thus produced were cylindrical in shape, with a diameter of 45mm and a height of ca. 20mm. Tablets with different densities were produced in order to determine the tablet disintegration and conductivity profiles. Tablets prepared had densities in the range 1250-1450 kg/m³, which are typical values for commercial fabric washing tablets found in the Western European market in 1998-9.

Determination of tablet disintegration profile

The tablet disintegration profile provides an indication of the extent to which various tablets (e.g. different compositions, different densities) disintegrate under the defined conditions.

4500 g of demineralised water at 20°C were added to a 5 litre vessel fitted with pH, conductivity and temperature probes and maintained at a constant temperature of 20°C by immersion in a water bath. The tablets to be tested are inserted into a metal cage having the dimensions 9cm x 4.7cm x 2.7cm and having 16 apertures (each about 2 mm square) per cm². The metal cage was attached to the shaft of an overhead stirrer (Heldolph/Janke and Kunkel stirrer) to allow it and its contents to be rotated while immersed in the demineralised water. Prior to testing, the empty cage was immersed in the demineralised water and rotated at 80 rpm for a short period of time until the temperature of the demineralised water as detected by the temperature probe had stabilised at 20 ± 0.2°C. At this time, the conductivity, pH and temperature values registered by the respective probes were recorded. The stirrer was then switched off to allow the cage to be raised out of the water so that a pre-weighed detergent tablet to be investigated could be inserted into the cage. The cage was then re-immersed in the demineralised water together with the inserted tablet and the stirrer was switched on to resume

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rotation of the container at 80 rpm. Measurements of conductivity and pH, initially at 15 second intervals for one minute and thereafter at one minute intervals, were made over a period of 10 minutes after which time the cage was raised out of the demineralised water to allow the residue of the tablet to be removed. The residue was then dried in an oven at 105°C together so that the dry weight of the residue could be calculated as a percentage of the original tablet weight. This procedure was repeated for a number of tablets having different compositions and different densities.

Determination of Tablet Conductivity Profile

The tablet conductivity profile provides an indication of the extent to which various tablets (e.g. different compositions, different densities) dissolve under the defined conditions. Conductivity measurements were obtained from the conductivity probe mentioned in the preceding paragraph at the same time as the tablet disintegration was measured. Measurements of conductivity and pH, were taken, initially at 30 second intervals for ten minutes and thereafter at one minute intervals, for a further 20 minutes or until the conductivity measurements were observed to have reached a plateau corresponding to substantially total dissolution of the soluble portion of the tablet, i.e. equilibration of the tablet with water. This procedure was repeated for a number of tablets having different compositions and different densities.

EXAMPLES

Conductivity and disintegration profiles were investigated for a number of tablet formulations and densities, all based on concentrated Persil (Registered Trade Mark) original non-Biological detergent powder as manufactured by Lever Brothers of Kingston-upon-Thames, UK - formulation as sold in the UK. The detergent powder was blended with different amounts of disintegrant granules. In each instance, the disintegrant granules comprised a water insoluble inorganic material, specifically a zeolite or a silica or a combination thereof, and a water-swellaable agent. For comparative purposes, granules comprising a mixture of a water soluble material, specifically sodium silicate or sodium carbonate, and a water swellaable material were also prepared and tested. In the Examples given below, the zeolite identified as A24 below was that sold under the trade mark Doucil A24 by Crosfield Limited of Warrington, UK; the silica identified as SD2255 is also obtainable from Crosfield Limited; the zeolite 4A is obtainable from Crosfield Limited under the trademark Doucil 4A, the sodium disilicate is obtainable from Crosfield Limited under the trade mark Pyramid 95; the sodium carbonate is obtainable from Solvay Chemicals Limited (identified in their product literature as Soda Ash (Sodium Carbonate) Light Rheinberg); and the water-swellaable agent was sodium carboxymethyl-cellulose as sold under the trade mark Aquasorb A500 by Hercules Limited of Salford, UK.

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TABLE 1

Property	SD2255	Doucl A24	Doucl 4A	Sodium Disilicate	Soda Ash	Aquasorb
Surface Area m ² /g	650	NM	NM	NM	NM	NM
Pore Volume ml/g	1.3	NM	NM	NM	NM	NM
APS microns	5	1.2*	3*	100*	See below	40*
Moisture Content %w/w	2	10*	20*	18-20*	< 1.5	5*
Oil absorption g/100g	228	60*	40*	NM	NM	NM

In Table 1, APS represents average particle size (d₅₀) as measured using a Malvern Mastersizer (Trade Mark) obtainable from Malvern Instruments in the UK and the values marked * are taken from typical data for the product or from specifications supplied by the manufacturer. The Soda Ash used in the Examples had a bulk density of 0.53 kg/dm³ and was found to have a particle size distribution (by sieve analysis) as follows:

- > 1000 microns = 1wt %
- 500 - 1000 microns = 0.5 wt %
- 250 - 500 microns = 3.5 wt %
- 75 - 250 microns = 74.5wt %
- 0 - 75 microns = 20.5wt %

The different formulations of the disintegrant granules (on a by weight basis) are given in Table 2.

CI 30828/ B

TABLE 2

Granule Code	Wt. % of Aquasorb A500 in granule	Remainder of Granule (Wt. % and Material)
A	10	90% SD2255 Silica
B	10	45% SD2255 Silica, 45% Doucil A24
C	10	90% Doucil 4A
D	10	90% Doucil A24
E	7.5	92.5% Doucil A24
F	5	95% Doucil A24
G	10	Pyramid 95
H	10	Sodium Carbonate

The granules coded G and H contain inorganic materials which are water soluble and as such are not within the scope of the present invention. These particular Examples are included for comparative purposes.

A series of tablets were been prepared at a standard density of $1325 \pm 10 \text{ kg/m}^3$. Tablets 1A-1H contain 2% by weight of granules A-H respectively, the other 98% being formed from concentrated Persil original non-Biological detergent powder. Tablet 1J was fabricated entirely from the same Persil powder. Tablets 1K and 1L were prepared from the same Persil powder, but incorporated 0.2% and 2% by weight of Aquasorb A500 powder respectively.

Table 3 shows the level of disintegration and the conductivity obtained after 10 minutes immersion in water for these tablets using the experimental protocol described above. The conductivity measurements are representative of the degree of dissolution of the soluble ionic constituents of the Persil detergent powder, the higher the conductivity value the greater the degree of tablet dissolution. The measurement of disintegration shows the level of undissolved residue retained in the 'cage'. Thus a high value indicates a poorly disintegrating tablet.

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TABLE 3

Tablet Code	Conductivity (microSiemen)	Undisintegrated Residue (%)
1A	3800	5
1B	4100	0
1C	4000	0
1D	3970	0
1E	3700	2
1F	3520	10
1G	1060	84
1H	2840	29
1J	1720	49
1K	1800	48
1L	630	80

The data clearly demonstrate the following:

- 1) The Tablet 1J, composed entirely of concentrated Persil powder does not disintegrate or dissolve well; after 10 minutes it is only 50% disintegrated.
- 2) Addition of 0.2% Aquasorb A500 (Tablet 1K) has no appreciable effect (this is equivalent to the amount of Aquasorb added via granule incorporation in Tablets 1A-1D, 1G and 1H).
- 3) Addition of more Aquasorb in Tablet 1L (equivalent to the total weight addition of granules in Tablets 1A-1H) actually appears to suppress disintegration and dissolution.
- 4) Tablets 1A-1D are essentially fully disintegrated/dissolved after 10 minutes.
- 5) In contrast, when the Aquasorb has been co-granulated with a soluble material (Tablets 1G, 1H), the disintegration and dissolution is far less effective. In the case of silicate, the granules actually appear to retard disintegration relative to Tablet 1J.
- 6) Even when the level of Aquasorb in the granules is reduced to 7.5% or 5% (Tablets 1E, 1F, disintegration of the Tablets is very well advanced after 10 minutes.

Table 4 below shows equivalent data for Tablets 2E, 2F. These contain granules E and F respectively, incorporated into tablets at 4% w/w instead of 2%. Again, it is clear that, by increasing the weight of granule in the tablet at these lower levels of Aquasorb inclusion, the excellent disintegrating properties of the granules can be retained without the need to significantly increase the total weight of disintegrating polymer in the Tablet (since 4% inclusion of granule F corresponds to 2% of granule D in terms of the total weight of Aquasorb introduced into the granule).

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TABLE 4

Tablet Code	Conductivity (microSiemen)	Undisintegrated Residue (%)
2E	3950	0
2F	3900	0

Table 5 below shows further data for disintegration/conductivity for tablets containing various of the granules, again incorporated into the tablets at 2%w/w. These tablets are identified as 3X, where the X is the letter corresponding to the granule identity in Table 2. In this case, the tablets have been prepared at a higher density ($1350 \pm 10 \text{ kg/m}^3$). At this higher density, it is possible to discriminate between the performance of the different granules A-D. From this data, it appears that the zeolite-containing granules are to be preferred over the silica variant, with the granules prepared from Doucil A24 being the best performing disintegrating agents.

TABLE 5

Tablet Code	Conductivity (microSiemen)	Undisintegrated Residue (%)
3A	3060	21
3B	3500	9
3C	3060	13
3D	3850	9

CE 30828/GB

CLAIMS

1. A cleaning composition, the composition including disintegrant in the form of granules comprising a water insoluble inorganic material and a water-swellaable agent which, in its anhydrous state, comprises no more than 20% of the combined weight of said inorganic material and said agent of the granular disintegrant, the granules being combined with the active ingredients of the cleaning composition in a compacted moulded body.
2. A cleaning composition in which the water-swellaable agent comprises, in its anhydrous state, no more than 15% of the combined weight of said inorganic material and said agent of the granular disintegrant.
3. A cleaning composition in which the water-swellaable agent comprises, in its anhydrous state, no more than 10% of the combined weight of said inorganic material and said agent of the granular disintegrant.
4. A cleaning composition in which the water-swellaable agent comprises, in its anhydrous state, no more than 8% of the combined weight of said inorganic material and said agent of the granular disintegrant.
5. A cleaning composition in which the water-swellaable agent comprises, in its anhydrous state, no more than 7.5% of the combined weight of said inorganic material and said agent of the granular disintegrant.
6. A cleaning composition as claimed in any one of the preceding claims in which the inorganic material comprises a silica.
7. A cleaning composition as claimed in any one of the preceding claims in which the inorganic material acts as functional ingredient of the cleaning composition.
8. A cleaning composition as claimed in any one of the preceding claims in which the inorganic material comprises an aluminosilicate.
9. A cleaning composition as claimed in Claim 8 in which the inorganic material comprises a zeolite.
10. A cleaning composition as claimed in Claim 9 in which the inorganic material comprises a zeolite P.
11. A cleaning composition as claimed in any one of Claims 7 to 10 in which the organic material comprises from about 10% to about 60% by weight of the cleaning composition.
12. A cleaning composition as claimed in Claim 11 in which at least 0.5% by weight of the inorganic material is embodied in the disintegrant granules.
13. A cleaning composition as claimed in any one of the preceding claims in which the moulded body consists entirely of said cleaning composition.
14. A cleaning composition as claimed in any one of Claims 1 to 13 in which only part of the moulded body is constituted by said cleaning composition, the remainder of the moulded body being constituted by at least one other ingredient for use in detergent and cleaning

CI 30828/GB

applications such as fabric washing, dishware washing, stain removal and water softening.

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ABSTRACT**CLEANING COMPOSITIONS**

A cleaning composition formed into moulded bodies for use in for example fabric washing, dishware washing, stain removal and water softening incorporates disintegrant granules comprising a water insoluble inorganic material and a water-swellaable agent which, in its anhydrous state, comprises no more than 20% of the combined weight of said inorganic material.

END

